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Measurements of the K X-ray intensity ratios by using energy-dispersive X-ray fluorescence spectrometry

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Abstract

K X-ray intensity ratios of some elements have been investigated by using radioisotope excited-energy-dispersive X-ray fluorescence spectrometer. The characteristic X-rays emitted from the samples have been recorded using a Si(Li) detector and they have been fitted to Gaussian functions by using a least-square method. The measured K X-ray intensity ratios have uncertainties of about 5%, and they are in good agreement with theoretical and experimental values available in the literature.

Keywords: Intensity ratio; EDXRF; Radioisotope

1. Introduction

K X-rays consists of mainly two groups, i.e. Kα and Kβ. Each K X-ray group occurs overlapped X-rays, namely Kα_1, Kα_2, and Kβ_1, Kβ_2, Kβ_3, Kβ_4, Kβ_5. These X-ray groups are listed in Table 1. The Kα X-rays are emitted when an electron transition occurs from L shell to K shell. If an electron is transmitted from M and higher shell to K shell, the Kβ X-rays are emitted. Deslattes et al. (2003) have evaluated the characteristic X-ray energies and their notation according to both Seigbahn and IUPAC.

The total rate of decay of a vacancy state of an isolated atom is the sum of the radiative and Auger transition rates. The fluorescence yield, i.e. the probability that a state decays accompanied by X-ray emission, is equal to the ratio of total X-ray emission rate to the total decay rate. The ratios of the intensities of the individual X-ray lines are equal to the ratios of the rates for the corresponding transitions (Scofield, 1974a).

Many measurements of the relative intensities of the K X-ray diagram lines, or sub-groupings of these lines, spanning almost the entire periodic table, have been reported for along time. The major objective of much of that work was generally to select which of the two theoretical treatments of relative K X-ray transition probabilities was the more appropriate, reflecting in part the need for an accurate database in elemental analysis techniques based upon X-ray emission spectroscopy. Both of these treatments were the work of Scofield, who employed successively Dirac–Hartree–Slater (Scofield, 1974a) and Dirac–Fock (DF) (Scofield, 1974b) atomic electron wavefunctions, allowing in latter case for the effects of overlap and exchange. Both treatments were free atoms (Campbell, 2001).

Knowledge of accurate intensity ratios is of great importance in X-ray emission techniques for elemental analysis. The measurements of K X-ray intensity ratios are important because by comparison with theoretical predictions based on atomic models, there is a possibility of testing the validity of these models (Ertuğrul et al., 2001). Because of this importance of intensity ratios, in this work, the K X-ray intensity ratios of some were investigated because of their importance in the many fields. The experimental results compared with the theoretically calculated results and some of the experimental results are available in literature.

2. Measurements

The measurements, as shown in Fig. 1, were carried out by an ^{241}Am radioisotope source, which emits 59.537 keV

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Table 1
K X-ray diagram lines and groups

Transitions		Sub-group	Group
K-L_2	Kα_1	Kα	Kα
K-L_3	Kα_2		
K-M_2	Kβ_3	Kβ'_1	Kβ
K-M_3	Kβ_1		
K-$M_{4,5}$	Kβ_5		
K-$N_{2,3}$	Kβ_2	Kβ'_2	
K-$N_{4,5}$	Kβ_4		

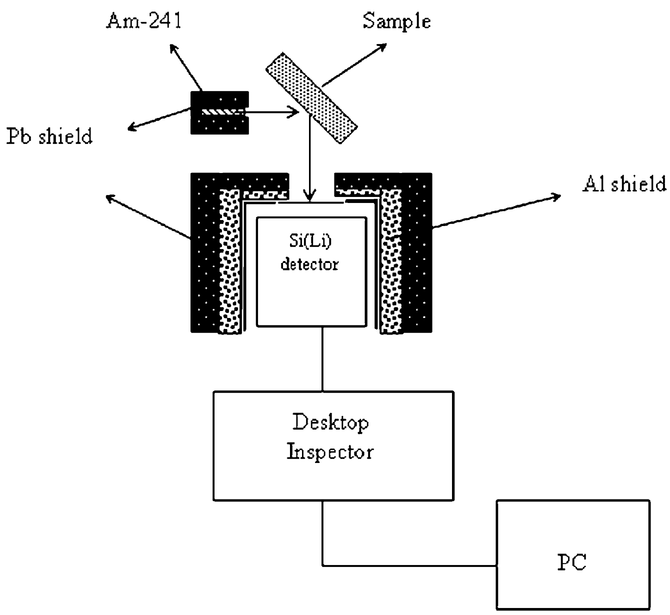

Fig. 1. Experimental set-up.

γ-rays; a Si(Li) detector connected to the Canberra desktop inspector. The desktop inspector includes an integrated MCA for detector, an amplifier, an analog digital converter (ADC), a high voltage power supply (HVPS), and a digital stabilizer. The Si(Li) solid-state detectors have 12.5 mm^2 active area and a resolution of 204 eV at 5.9 keV Mn Kα line, and a sample. The direct beams (59.537 keV γ-rays) from the source mentioned above were incident on the sample and the secondary X-rays emitted from the sample were recorded at the Si(Li) detector. In the present experimental set-up, the angles between incident and emitted X-ray beams normal to the sample surface are set to 45°. The measurement time was 3 h for each sample. Some of the samples, Cr, Fe, Co, Ni, Cu, Zn, Gd and Dy, have been in foil form. The foils have been cut into a sphere shape with 0.65 mm. The other samples, KBr, Nb_2O_5, BaO, Nd_2O_3, $SmCl_3$, $EuCl_2$, $HoCl_3$, have been in powder form. They are sieved to minimize the particle size effects with a sieve (200 meshes). They were made into pellets of uniform thickness with a Beckman press. These pellets have 0.65 cm radius and possibly less thickness. Two recorded full spectra of Ni and Dy are illustrated in the Fig. 2.

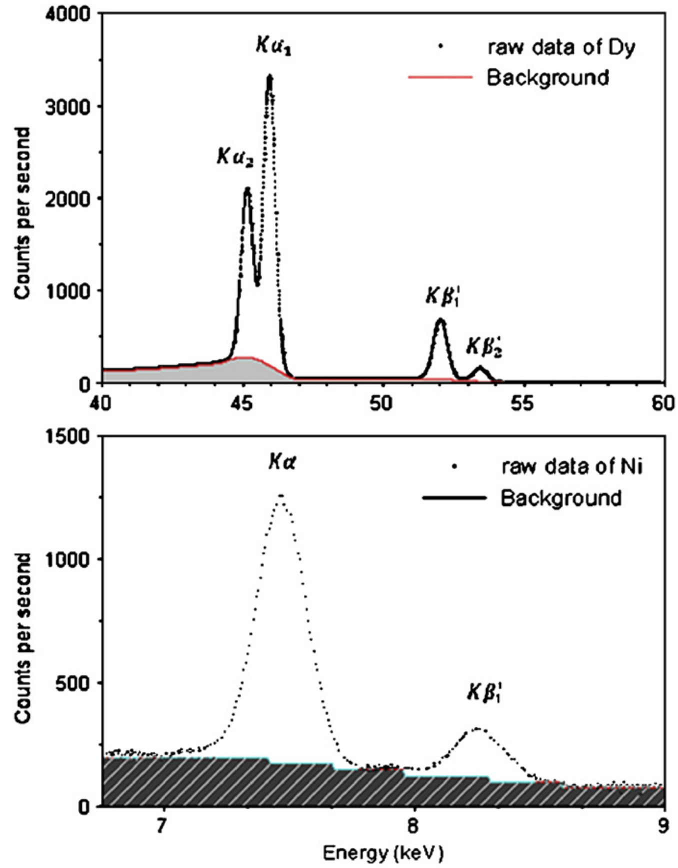

Fig. 2. Typical spectra of Ni and Dy. In this figure, both raw data of samples and background determined according to the method of Şahin et al. (1996) are illustrated.

3. Data analysis procedure

The Kx_i/Kα_1 intensity ratio values have been calculated using the relation (Ertuğrul et al., 2001)

$$\frac{I(\mathrm{K}x_i)}{I(\mathrm{K}\alpha_1)} = \frac{N(\mathrm{K}x_i)}{N(\mathrm{K}\alpha_1)} \frac{\varepsilon(\mathrm{K}\alpha_2)}{\varepsilon(\mathrm{K}x_i)} \frac{\beta(\mathrm{K}\alpha_1)}{\beta(\mathrm{K}x_i)} \quad x_i = \alpha_2, \beta_1, \beta_2,$$

where $N(\mathrm{K}\alpha_1)$ and $N(\mathrm{K}x_i)$ are counts under the peaks corresponding to Kα_1 and Kx_i X-rays, respectively, $\varepsilon(\mathrm{K}\alpha_1)$ and $\varepsilon(\mathrm{K}x_i)$ are the efficiencies of the detector for the Kα_1 and Kx_i X-rays, respectively (Budak et al., 1999). $\beta(\mathrm{K}\alpha_1)$ and $\beta(\mathrm{K}x_i)$ are the target self-absorption correction factors for incident and emitted radiation and they are given by

$$\beta = \frac{1 - \exp\{-[\mu_{inc} \sec\theta_{inc} + \mu_{emit} \sec\theta_{emit}]\}}{[\mu_{inc} \sec\theta_{inc} + \mu_{emit} \sec\theta_{emit}]},$$

where μ_{inc} and μ_{emit} are the mass-attenuation coefficients of the target at the incident photon energy (59.537 keV) and Kα_1 and Kx_i X-rays' energies, respectively. These coefficient values have been taken from the Windows version of the XCOM (Berger and Hubbell, 1999), namely